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## NDE of Ceramics and Ceramic Composites

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## NDE OF CERAMICS AND CERAMIC COMPOSITES

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### SUMMARY

Although NDE techniques for ceramics are fairly well-developed they are difficult to apply in many cases for high probability detection of the minute flaws that can cause failure in monolithic ceramics. Conventional NDE techniques are available for monolithic and fiber reinforced ceramic matrix composites, but more exact quantitative techniques needed are still being investigated and developed. Needs range from flaw detection to below 100  $\mu\text{m}$  levels in monolithic ceramics to global imaging of fiber architecture and matrix densification anomalies in ceramic composites. NDE techniques that will ultimately be applicable to production and quality control of ceramic structures are still emerging from the laboratory. Needs are different depending on the processing stage, fabrication method, and nature of the finished product. NDE techniques are being developed in concert with materials processing research where they can provide feedback information to processing development and quality improvement. NDE techniques also serve as research tools for materials characterization and for understanding failure processes, e.g., during thermomechanical testing.

### INTRODUCTION

Silicon carbide and silicon nitride are leading candidate materials for hot section components in advanced terrestrial automotive and aerospace heat engines (ref. 20). Although having good to average high temperature strength and excellent oxidation resistance, the brittle nature and acute sensitivity of monolithic ceramics to minute defects leads to wide variations in mechanical properties and relatively low fracture toughness (ref. 31). Improvements in strength and toughness can be achieved by reinforcement with ceramic fibers to produce ceramic matrix composites. While the strength of monolithic ceramics is generally governed by the size, population, and distribution of defects, fiber reinforced ceramics are relatively insensitive to the size of pre-existing flaws in the matrix (ref. 13). The strength, toughness, and fracture resistance of ceramic matrix composites depend primarily on the fiber strength, the degree of fiber-matrix interface bonding, and the ability of the matrix to distribute and absorb fracture energy.

Advanced structural ceramics for high temperature applications in space power and propulsion systems pose challenges for NDE that exceed those encountered with engineering metals (refs. 1 and 40). This is particularly true of ceramic composites with structural properties that are tailored and created in place during processing stages (ref. 24). Special NDE methods are required for inspecting powders and green compacts before monolithic ceramics are densified by hot pressing or sintering (ref. 19). In fully densified finished components NDE techniques must

certainly detect and characterize various types of discrete defects like cracks, voids, and other overt discontinuities. But it is equally important to detect and characterize microstructural and diffuse flaw conditions that govern overall strength, fracture toughness, impact resistance, and resistance to thermal-mechanical-chemical degradation. These diffuse flaw states can reduce reliability and diminish service life just as much as discrete flaws (ref. 38).

The emphasis in this section is on NDE techniques applicable to fully densified and finished monolithic ceramic and ceramic composite articles. However, all the techniques mentioned should be considered for use during processing to ensure quality, during fabrication to screen out defective pieces, and during service to assess any chemical, thermal, or mechanical damage.

## MONOLITHIC AND TOUGHENED CERAMICS

For monolithic ceramics the problem is to detect minute flaws such as cracks, voids, grain agglomerations, and foreign inclusions having sizes to below the 100  $\mu\text{m}$  level (ref. 22). Porosity, diffuse flaw populations, texture and density variations need to be found for their potentially deleterious effects on the strength and fracture resistance of monolithic and toughened ceramics. Flaw detection must deal separately with surface, near-surface, and volume flaws. In addition to discrete, isolated "dominant" flaws (cracks, agglomerates, voids) it is necessary to characterize the microstructure relative to diffuse porosity variations, density gradients, or grain size fluctuations. These latter conditions form the environments of discrete flaws and determine susceptibility to crack growth and fracture. In the case of particulate, transformation, and whisker toughened ceramics microstructural anomalies, composition and density variations, and adverse textures (e.g., nonrandom whisker alignments) need to be detected (refs. 28 and 32).

## CERAMIC MATRIX COMPOSITES

Ceramic matrix composites can be formed as continuous fiber composite laminates or as two and three-dimensional woven composites. Ceramic composites must be inspected for delaminations and other overt discontinuities as well as for harmful local and global variations in matrix densification, fiber distribution, fiber architecture, intralaminar integrity, and fiber-matrix bond quality (ref. 30). Laminated and woven ceramic matrix composites present totally different challenges to NDE than do monolithic and toughened ceramics. Ceramic composites need to be approached from the viewpoint that the detection of individual microflaws is unnecessary. This does not mean that large overt flaws such as delaminations, cracks, and similar discontinuities can be ignored. Instead, it should be recognized that composites may contain a profusion of minute defects that have no discernable effect on reliability or performance unless they are in close proximity and interact massively. Therefore, what must be detected are aggregates of flaws that can collectively degrade reliability and performance. Sparsely distributed, occasional matrix cracks, broken fibers, or misaligned fibers need be of little concern. But, generally poor bonding between fiber and matrix must be of high concern (ref. 13). This raises the issue of the strength of fiber-matrix interfaces and interphases that will determine fracture toughness and impact resistance. The NDE problem then becomes one of characterizing the collective effect of fiber-matrix bonds on mechanical quality and strength in a volume of material. This is, of course, always in addition to the need to detect any overt, dominant discontinuities that would have an overriding effect on structural integrity under monotonic loading, fatigue, or impact.

## NDE TECHNIQUES

General NDE techniques applicable to ceramics and their composites are optical examination, liquid penetrant inspection, radiography, and ultrasonics (ref. 19 and 40). Specialized techniques include microfocus x-radiography, computed tomography, analytical ultrasonics, and acoustic microscopy for monolithic and particulate and whisker toughened ceramics. Computed tomography, film and digital radiography, scanning ultrasonics, and acousto-ultrasonics are among the specialized NDE techniques suitable to inspecting ceramic fiber reinforced ceramic matrix composites.

### Conventional Methods

Conventional, appropriate, and mandatory NDE techniques for surface-connected flaws are optically aided visual and liquid penetrant inspections (ref. 10). They should be used routinely to screen out articles that are cracked, pitted, marred, spalled, or have poorly finished surfaces. Immersion scanning ultrasonics, film radiography, and computed tomography are required to detect subsurface and volume flaws. If the flaws are isolated and fairly large, i.e., of the order of 500  $\mu\text{m}$  or more, then conventional ultrasonic scanning and film radiographic methods are quite suitable. However, the spatial and image density resolution of these conventional methods becomes taxed in the "grey area" represented by flaws in the 500 to 50  $\mu\text{m}$  size range.

### High Resolution Methods

For discrete flaws below 100  $\mu\text{m}$  in size it is certainly necessary to consider high resolution methods like acoustic microscopy and microfocus radiography and tomography. Acoustic microscopy and microfocus radiography methods have been shown to resolve flaws down to the 20  $\mu\text{m}$  level in monolithic silicon carbide and silicon nitride. But these methods are successful and have high probability-of-detection only under the most stringent conditions of material thickness, surface finish, etc. (refs. 5, 23, and 27). But even under the best conditions and with high resolution NDE, some flaws remain very difficult to detect, e.g., tight cracks, megagrains, and agglomerates having densities or acoustic impedances that match their background.

### Materials Characterization

In monolithic ceramics flaws less than 10  $\mu\text{m}$  in size have been routinely found to be fracture origins. Such flaws tend to be quite numerous in fine-grained ceramics and this situation will overburden the capabilities of any high resolution NDE technique. High resolution imaging is inherently time-consuming. Given this situation, it is important to decide whether there is sufficient pay-off to examine each and every cubic millimeter of a monolithic ceramic article for each 10  $\mu\text{m}$  flaw. Below the 50  $\mu\text{m}$  level it may be impractical and even unnecessary to image and characterize individual flaws in a monolithic ceramic. The best alternative is that of characterizing the microstructural environment in which such flaws reside (refs. 6 and 36). This is the primary goal of analytical ultrasonics and computed tomography. These two methods, discussed later in this section, are needed to quantify and image diffuse flaw states as represented by porosity distributions, nonrandom whisker alignments, and similar local or global anomalies.

## Acoustic Microscopy

Fairly large flaws are frequently encountered in components such as turbine rotors. But, recent laboratory results on new high strength monolithic ceramics have shown that many failures are initiated by surface and near-surface defects between 20 and 40  $\mu\text{m}$  in size. Acoustic microscopy affords the potential for detection of flaws of this nature, given the right conditions. Surface preparation by polishing or fine grinding is needed to enhance the detectability even of exposed surface voids on the order of 50  $\mu\text{m}$  diameter. Surface roughness affects the signal-to-noise ratio in acoustic microscope images. Moreover, sintered samples with as-fired surfaces show decreased volume flaw detectability with increased thickness. Flaw detectability also depends on the relative coarseness of the material microstructure. In silicon carbide samples flaw detectability was found to be significantly less than in silicon nitride samples that had a much finer grain (ref. 23).

Scanning acoustic microscopy. - Scanning acoustic microscopy (SAM) is a pulse reflection ultrasonic technique that uses a single spherically focused transducer (ref. 25). The test article must be immersed or coupled with a liquid medium. By adjusting the distance between the transducer and test object it is possible to place the focal point at the desired plane in the object. Precision mechanical scanning is used to build images of features in the vicinity of the plane of interest. Typical scans, covering an area of roughly 15  $\text{mm}^2$  utilize relatively slow mechanical microscanners and may take several minutes to develop an image with a resolution of about 20  $\mu\text{m}$ . SAM devices usually operate at 50 to 200 MHz and can be focused up to several millimeters into fine grained monolithic ceramics. A SAM device operating at a center frequency of 50 MHz is readily able to image voids 20  $\mu\text{m}$  in diameter at a depth of 1 mm in silicon nitride (ref. 39).

Surface wave method. - Near surface flaws may require examination by the surface wave method (ref. 26). A focused ultrasonic transducer operating at frequencies up to 100 MHz is used to launch and collect Rayleigh waves that can interact with and resolve minute cracks and other defects down to the 10  $\mu\text{m}$  level. The surface wave method overcomes difficulties encountered by the pulse reflection SAM method, mentioned previously, primarily because the waves travel parallel rather than normal to the surface.

## Analytical Ultrasonics

The term analytical ultrasonics denotes NDE methodology for quantitative characterization of the microstructure and mechanical properties of engineering materials (ref. 35). Ultrasonic velocity and attenuation have been successfully correlated with bulk density, grain size, strength, and toughness (ref. 37). Models explaining and predicting the empirical correlations between ultrasound and mechanical properties have been advanced (ref. 36). Accurate determination of these correlations depends on the experimental technique used to make the ultrasonic measurements. Factors that influence ultrasonic attenuation and velocity measurements include surface finish, pore fraction, pore size and shape, grain size, grain size distribution, texture, and elastic anisotropy. Of course, these same factors also govern mechanical properties and response to service conditions.

Attenuation measurements. - The effect of grain size and porosity on attenuation of specially prepared silicon carbide specimens is shown in figure 1. Three batches of material were sintered under different conditions to produce samples with varying bulk density and mean pore

size with corresponding variations in grain size (ref. 7). Low ultrasonic attenuation is characteristic of nearly fully dense monolithic ceramics with fine microstructures, i.e., samples with densities greater than about 95 percent of theoretical. For monolithic and toughened ceramics significant attenuation differences are evident only at frequencies greater than approximately 100 MHz where the ultrasonic wavelength is less than 10 to 20 times the average grain and pore size. Ultrasonic attenuation is influenced by bulk density and the combined effects of pore size and grain size and, therefore, is a sensitive indicator of microstructural variations in structural ceramics when measurements are made at the appropriate frequencies (ref. 15). However, meaningful attenuation measurements require not only fairly smooth surfaces but also constraints on sample size, shape, and thickness. When accurate attenuation measurements are needed, the surface roughness should not exceed peak-to-valley heights of 1  $\mu\text{m}$  (ref. 14). Nevertheless, it is possible to make comparative attenuation measurements on specimens with rougher surfaces as long as the finish is the same on all samples.

Velocity measurements. - Velocity is a monotonically increasing function of density in most porous solids. Variations in pore size and grain size have little effect on this correlation. Figure 2 shows velocity data for nearly 200 silicon carbide specimens in either the as-sintered or hot-isostatically pressed conditions. The specimens exhibited a wide range of grain sizes and shapes as well as variations in pore morphology and distribution (ref. 21). Velocity measurements can be used to screen out specimens and components with low densities. Although variations in surface finish and overall sample thickness can reduce accuracy somewhat, velocity measurements are not as vulnerable to surface roughness as are attenuation measurements. Experimental results show that velocity measurements can be used to estimate bulk density within approximately one percent. Because velocity measurements are not strongly affected by pore or grain size, they are convenient for estimating bulk density of monolithic and toughened ceramics, even though velocity measurements are generally less sensitive than attenuation measurements.

Backscatter ultrasonics. - Both attenuation and velocity measurements require essentially flat, parallel opposing surfaces or geometric simplicity. Part shapes are not always amenable to precision attenuation or velocity measurements. An alternative approach is that of utilizing the backscatter method for ultrasonic determination of porosity, grain, and similar microstructural variables (ref. 18). Backscattered, and under some conditions forward scattered, ultrasound radiations can be used to characterize volume properties of parts having complex shapes.

Acousto-ultrasonics. - The acousto-ultrasonic (A-U) technique was developed specifically for characterizing defect states and mechanical property variations of composites (ref. 11). Two piezoelectric transducers coupled to the surface of the test sample are used in a send-received mode. The A-U technique has been applied to fiber reinforced composite laminates to detect local and global anomalies such as matrix crazing and porosity, modulus or stiffness variations, and fatigue damage. The technique measures efficiency of in-plane wave propagation in laminates and is similar to coin tap, sonic vibration, and dynamic resonance methods for assessing the overall condition of fabricated shapes (refs. 2 and 34). The A-U technique is a comparative analytical ultrasonic method that does not impose the stringent constraints on material surface conditions required for the attenuation measurements, mentioned previously.

## Radiography

Film radiography and projection radiography are important NDE imaging methods for flaw detection, for assessing the uniformity of density, and for locating porosity in monolithic and toughened ceramics (ref. 12). Although conventional versions of the methods are qualitative they provide excellent means for comparing varying degrees of densification in a volume of material. Figure 3 shows radiographs of three silicon nitride test bars with different mean bulk densities. One bar was near theoretical density (bottom) while the other two exhibited density gradients characterized by a core with relatively high porosity (light zones) and decreasing pore fraction toward the surfaces (darker regions). These results, when fed back to materials researchers, resulted in improved sintering methods that produced samples with uniform densities near theoretical and a 60 percent increase in strength (ref. 28).

Microfocus Radiography. - Microfocus radiography provides a high resolution imaging tool with the potential of being readily applied in production as well as laboratory environments. Film and real-time video versions are available for inspecting a variety of test objects for flaws distributed throughout a volume. Recent research has shown the combined spatial and image density resolution of microfocus radiography to be at least twice that of conventional film radiography (refs. 4 and 5). Like other projection radiographic methods, microfocus radiography is suitable for detecting flaws that have three-dimensional extent, as opposed to those that are two-dimensional or planar, like cracks.

Computed Tomography. - Computed tomography (CT) systems can produce the high resolution images required for characterization of structural ceramics and their composites (ref. 17). Unlike film and projection radiography, CT produces cross-sectional and three-dimensional reconstructions of both discrete and diffuse flaw populations in an examined volume. Flaw location and characterization of internal structures are easy with CT. High speed industrial CT systems readily provide image resolutions on the order of 250  $\mu\text{m}$ . Using microfocus x-ray sources, advanced CT systems are being developed for resolving down to 25  $\mu\text{m}$  (ref. 3). An example of preliminary results is shown in figure 4. The image is a CT cross section of a small SiC/Ti composite sample. The silicon carbide fibers are 140  $\mu\text{m}$  in diameter. The center of the fibers is composed of a carbon core which is only 25  $\mu\text{m}$  in diameter and is seen as a dark spot in the middle of each fiber. However, the estimated image resolution of the current prototype system is approximately 50  $\mu\text{m}$ . The potential for high resolution flaw detection is not the only benefit to be gained from CT. Another important benefit is realized by using CT at lower resolutions to record and reconstruct three-dimensional images of density variations, fiber architecture, dispersed flaw populations, and similar global features in structural ceramic components.

## NDE AND FRACTURE ANALYSIS

Constitutive modeling and fracture analysis should be used in concert with NDE to specify critical flaw conditions and to define inspection sensitivity requirements. The objective should be to set foundations upon which NDE can base assessments of integrity and service life (Refs. 13 and 24). Meeting this objective entails the conduct of research for validating various failure prediction models. The research should incorporate NDE techniques before and during mechanical destructive testing to establish inspection procedures that can identify critical defect states. These defect states should include not only overt flaw populations but also detrimental microstructural and morphological background conditions.

The conventional approach to in situ NDE monitoring of initial fracture and damage accumulation is acoustic emission. A recently proposed approach combines computed tomography with constitutive modelling prior to destructive testing (ref. 33). This approach would use CT images to measure and reconstruct, say, density variations in a test article and translate the data into a finite element analysis model to predict stress and strain states under specific loading conditions. The idea is to combine NDE methodology and fracture analysis for understanding the effects of different defect states.

## NDE AND PROCESS CONTROL

The structural integrity of monolithic ceramics and ceramic composites depends on avoiding fabrication flaws and maintaining high quality during processing (ref. 9). An approach to consistently producing high quality ceramics is to utilize NDE techniques during materials research and processing development to help determine stages when harmful flaws are likely to be introduced (ref. 16). Steps can then be taken minimize their occurrence through improvements in processing. Another approach is to use NDE as a materials characterization tool to assure that components possess correct microstructures and uniform properties. This can be done at various stages of processing to save the cost of finishing parts that contain defects from an earlier stage. The least efficient approach, although usually unavoidable, is to use NDE after the last stage of fabrication to eliminate parts that contain harmful flaws.

## STATE OF THE ART

In general, materials characterization and high resolution flaw detection methods like acoustic microscopy are laboratory techniques that require further investigation, development, and adaptation before they can be applied in materials processing, fabrication, and field environments. Practical realization of these methods for production and field use awaits the development of suitable calibration standards and standards of practice (ref. 8). Flaw detection techniques for monolithic and toughened ceramics depend on investigations that will establish statistical foundations for probability-of-detection of various types of defects over a range of material and component conditions (refs. 4 and 23). Material characterization techniques for ceramics and ceramic composites are new approaches that require careful investigation and development before they can be relied on to assess overall defect states or degrees of thermo-mechanical degradation.

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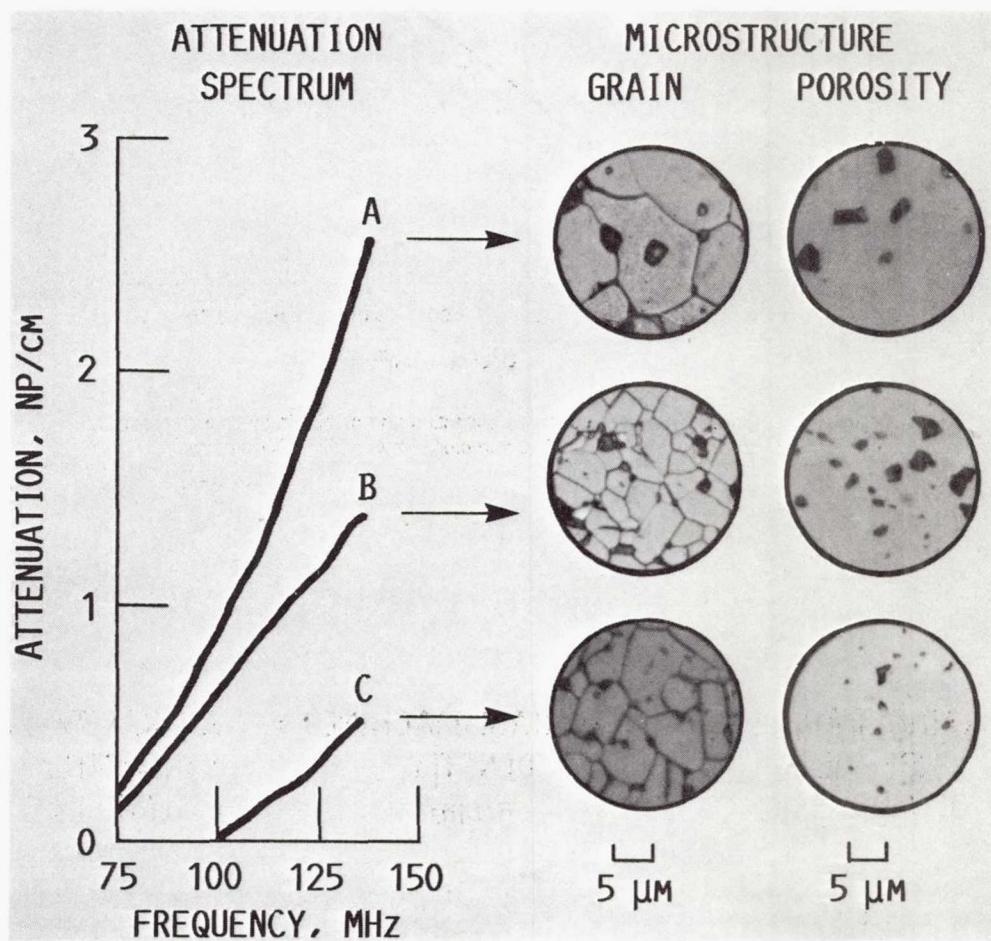


Figure 1.—Correlation of ultrasonic attenuation spectrum with grain and porosity variations among monolithic silicon carbide samples.

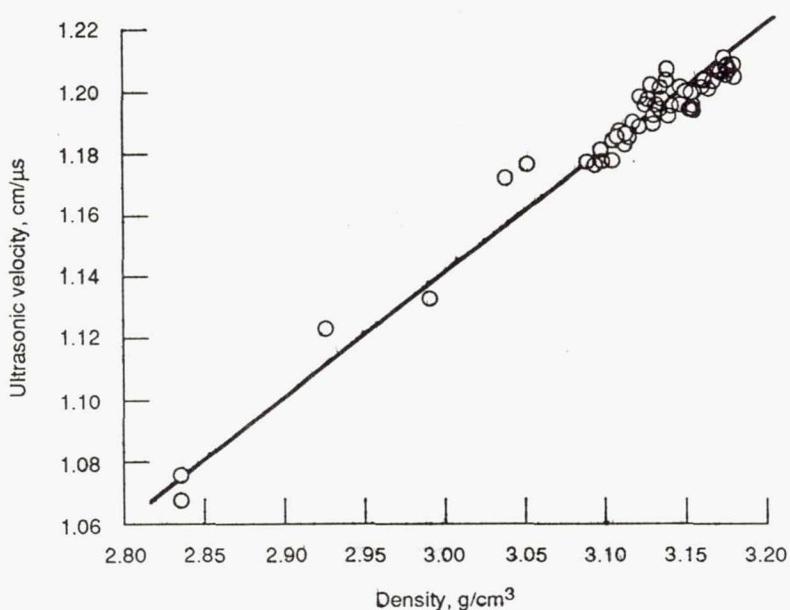


Figure 2.—Composite curve collecting and combining velocity versus density data for over 200 samples of monolithic silicon carbide.

RADIOGRAPHIC SIDE VIEW OF MOR BAR	AVERAGE MACHINED DENSITY, g/cm <sup>3</sup>	AVERAGE FLEXURAL STRENGTH AT ROOM TEMPERATURE, MPa
	3.12	548
	3.24	632
	3.24	868

Figure 3.—Effect of sintering variables on porosity distribution and uniformity of density as revealed by x-radiography.

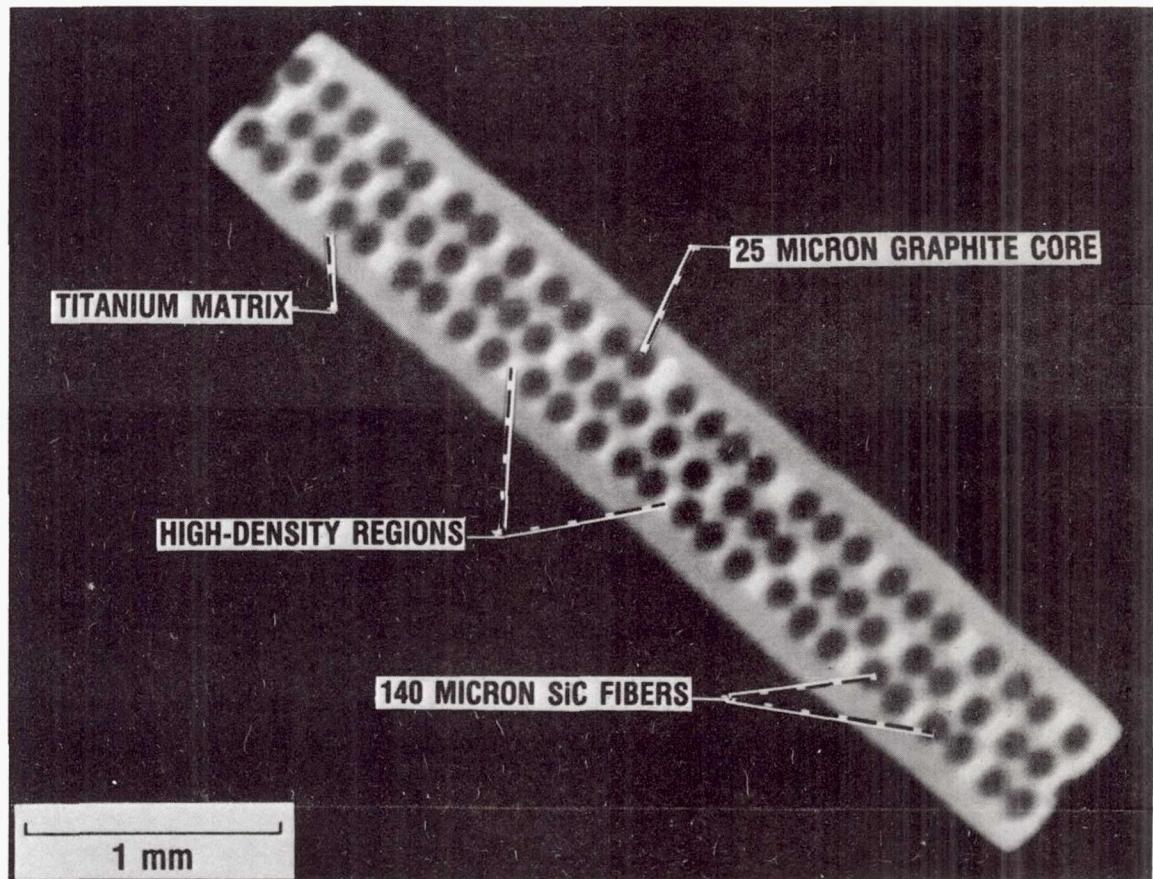


Figure 4.—High resolution computed tomographic cross-section image of ceramic fiber reinforced metal matrix composite sample.



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